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Ethyl 3-methyl-6-oxo-5-[3-(trifluoromethyl)phenyl]-1,6-dihydro-1-pyridazineacetate

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Key indicators

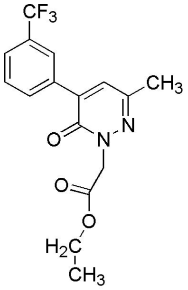
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.185
Data-to-parameter ratio = 12.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{16}H_{15}F_3N_2O_3$, is composed of a 3-(trifluoromethyl)phenyl ring and a pyridazinone ring, which are approximately coplanar, and an ethyl acetate group on the pyridazinone ring. In the crystal structure, centrosymmetrically related molecules form dimers through non-classical intermolecular C—H···O hydrogen bonds.

Comment

The carotenoid biosynthetic pathway is an important site of herbicide activity. Heterocyclic compounds, such as norflurazon, flurochloridone or diflufenican, prevent the formation of carotenoids and some of them have already been commercialized as herbicides (Ohki *et al.*, 2003). Most of these herbicides belong to different chemical classes, but they have a common substituent, *viz.* a 3-(trifluoromethyl)phenyl group. This led us to study the syntheses and structures of these compounds. To further examine the relationship between the structure and herbicidal activity, we synthesized a series of pyridazinone derivatives containing a 3-(trifluoromethyl)phenyl group. We report here the crystal structure analysis of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond distances and angles are given in Table 1. The 3-(trifluoromethyl)phenyl and pyridazinone rings are approximately coplanar with a dihedral angle of 4.84 (13)°. The conformation of the attachment of the benzene ring to the pyridazinone ring is described by the torsion angle C1—C2—C6—C7 of 175.5 (3)°.

In the crystal structure of (I), centrosymmetrically related molecules form dimers through non-classical intermolecular C—H···O hydrogen bonds. This situation is illustrated in Fig. 2 and details of the hydrogen bonding are given in Table 2.

Experimental

The title compound, (I), was synthesized according to the procedure reported by King & McMillan (1952). A mixture of 6-methyl-4-[3-

(trifluoromethyl)phenyl]pyridazin-3(2*H*)-one and ethyl bromoacetate in sodium ethoxide solution was stirred at room temperature for about 2 h. The organic phase was concentrated *in vacuo*, and the resulting residue was purified by column chromatography. After removing the eluant, the solid was recrystallized from a mixture of chloroform and hexane, giving single crystals suitable for X-ray diffraction.

Crystal data

$C_{16}H_{15}F_3N_2O_3$
$M_r = 340.30$
Monoclinic, $P2_1/n$
$a = 7.4846\ (14)$ Å
$b = 8.1059\ (15)$ Å
$c = 27.139\ (5)$ Å
$\beta = 97.867\ (4)^\circ$
$V = 1631.0\ (5)$ Å^3
$Z = 4$

$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1728 reflections
$\theta = 2.6$–21.2°
$\mu = 0.12$ mm^{-1}
$T = 294\ (2)$ K
Block, colorless
$0.32 \times 0.28 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.952$, $T_{\max} = 0.988$
8917 measured reflections

3339 independent reflections
1552 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\max} = 26.4^\circ$
$h = -7 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.185$
$S = 1.00$
3339 reflections
260 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2 + 0.5612P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.363 (3)	C6—C7	1.392 (4)
N1—C1	1.381 (4)	C6—C11	1.396 (4)
N2—C4	1.299 (4)	C7—C8	1.376 (4)
C1—C2	1.452 (4)	C8—C9	1.379 (5)
C2—C3	1.360 (4)	C8—C12	1.466 (5)
C2—C6	1.487 (4)	C9—C10	1.363 (5)
C3—C4	1.408 (4)	C10—C11	1.376 (5)
C4—C5	1.506 (4)		
N2—N1—C1	127.4 (2)	C7—C6—C2	120.0 (3)
N2—N1—C13	114.9 (3)	C11—C6—C2	123.1 (3)
C3—C2—C1	116.2 (3)	C8—C7—C6	121.6 (3)
C3—C2—C6	121.9 (3)	C7—C8—C9	120.5 (3)
C1—C2—C6	121.9 (3)	C7—C8—C12	119.1 (4)
C2—C3—C4	122.8 (3)	C9—C8—C12	120.4 (4)
N2—C4—C3	122.4 (3)	C10—C9—C8	118.5 (4)
N2—C4—C5	117.5 (3)	C9—C10—C11	121.8 (4)
C3—C4—C5	120.1 (3)	C10—C11—C6	120.6 (3)
C7—C6—C11	116.9 (3)		
C1—N1—N2—C4	−0.4 (4)	C3—C2—C6—C11	176.5 (3)
C13—N1—N2—C4	179.4 (2)	C1—C2—C6—C11	−4.1 (4)
C13—N1—C1—C2	178.6 (2)	C11—C6—C7—C8	−0.2 (4)
N1—C1—C2—C3	2.7 (4)	C2—C6—C7—C8	−179.9 (3)
N1—C1—C2—C6	−176.7 (2)	C6—C7—C8—C9	0.4 (5)
C1—C2—C3—C4	−1.9 (4)	C6—C7—C8—C12	−179.4 (3)
C6—C2—C3—C4	177.5 (2)	C7—C8—C9—C10	−0.3 (5)
N1—N2—C4—C3	1.4 (4)	C12—C8—C9—C10	179.5 (4)
N1—N2—C4—C5	−178.9 (2)	C8—C9—C10—C11	0.1 (6)
C2—C3—C4—N2	−0.3 (4)	C9—C10—C11—C6	0.0 (6)
C2—C3—C4—C5	−179.9 (3)	C7—C6—C11—C10	0.0 (5)
C3—C2—C6—C7	−3.8 (4)	C2—C6—C11—C10	179.6 (3)
C1—C2—C6—C7	175.5 (3)		

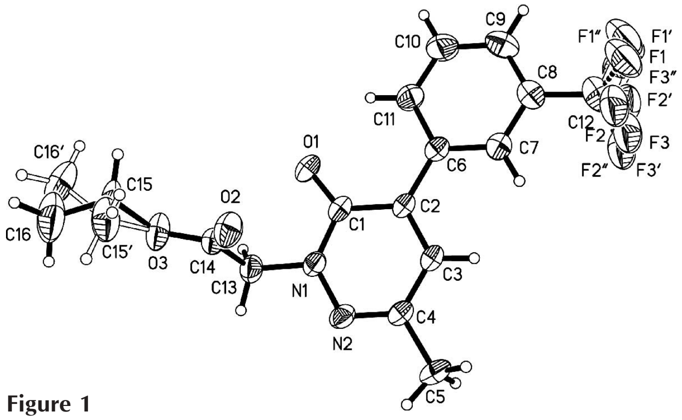


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme. All disorder components are shown.

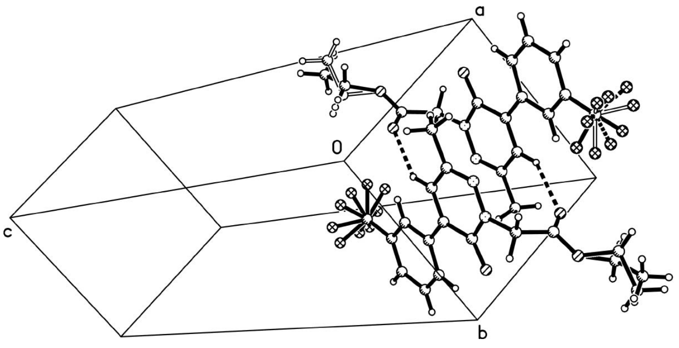


Figure 2
A partial view of the crystal packing in (I), showing the non-classical C—H···O hydrogen-bonded dimers. The intermolecular hydrogen bonds are shown as dashed lines. All disorder components are shown.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2$^{\rm i}$	0.93	2.56	3.395 (4)	150
C11—H11···O1	0.93	2.19	2.847 (4)	127

Symmetry code: (i) $1 - x, 1 - y, -z$.

The trifluoromethyl group was disordered over three positions, with occupancies 0.452:0.297:0.251. Atoms C15 and C16, of the ethyl acetate substituent are disordered over two positions, with occupancies 0.52:0.48. H atoms were placed in calculated positions and included in the final cycles of refinement using a riding model, with C—H = 0.93 or 0.97 Å, N—H = 0.86 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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